भारतीय मानक
Indian Standard

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कोयले, कोक, लिग्नाइट एवं उड़न राख में दस अल्प तत्वों को ज्वाला आण्विक अवचूषण स्पेक्ट्रोस्कोपिक पद्धति के द्वारा ज्ञात करना

Determination of Ten Trace Elements in Coal, Coke, Lignite and Fly Ash by Flame Atomic Absorption Spectroscopic Method

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Solid Mineral Fuels and Solid Biofuels Sectional Committee, PCD 07

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Solid Mineral Fuels and Solid Biofuels Sectional Committee had been approvedby the Petroleum, Coal and Related Products Division Council.

A wide range of trace elements occur in coal, coke and lignite. Their presence in coal and coal combustion products assume significance due to their essentiality, non-essentiality, toxicity and related environmental issues. Concern over release of these elements to the environment as a result of combustion, disposal of wastes and utilization of combustion by-products in agriculture as soil modifier and source of micronutrients has made the determination of trace elements an important aspect of coal, coke, lignite and ash analysis.

In the formulation of this standard, due weightage has been given to the international co-ordination among the standards and practices prevailing in different countries in addition to the practices in India and considerable assistance derived from the following publications:

AS 1038.10.1-2003	Coal and coke — Determination of trace elements — Determination of eleven trace elements in coal, coke and fly ash — Flame atomic absorption spectrometric method
ASTM D3683-11	Standard test method for trace elements in coal and coke ash by atomic absorption
ISO 23380 : 2013	Selection of methods for the determination of trace elements in coal

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

DETERMINATION OF TEN TRACE ELEMENTS IN COAL, COKE, LIGNITE AND FLY ASH BY FLAME ATOMIC ABSORPTION SPECTROSCOPIC METHOD

1 SCOPE

This standard covers sample preparation, dissolution and flame atomic absorption spectroscopic method for the estimation of Cadmium, Chromium, Cobalt, Copper, Manganese, Nickel, Lead, Lithium, Vanadium and Zinc in coal, coke, lignite and fly ash.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibilities of applying the most recent editions of the standards.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
265 : 1993	Hydrochloric acid — Specification (*third revision*)
436 (Part 1/ Sec 1) : 1964	Methods for sampling of coal and coke: Part 1 Sampling of coal, Section 1 Manual sampling(*revised*)
436 (Part 2): 1965	Methods for sampling of coal and coke: Part 2 Sampling of coke (*revised*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1350 (Part 1): 1984	Methods of test for coal and coke: Part 1 Proximate analysis (*second revision*)
6491 : 1972	Methods of sampling fly ash
10073 : 1982	Plastics graduated measuring cylinders
10332 : 1982	Specification for hydrofluoric acid, aqueous

3 PRINCIPLE

Representative sample of coal, coke, ligniteor fly ash isashed under controlled laboratory conditions. The resulting ash is dissolved in mineral acids and the individual elements are determined by flame atomic absorption spectrometry.

4 REAGENTS

4.0 General

Reagents used in preparation of standard stock solution for each element shall be pure and free from elements to be determined. Stock solutions of 1 000 ppm and 100 ppm of metal ions shall be stored in polythene/PTFE bottles with leak proof screw caps and labelled with the date of preparation. The stock solution shall not be used any longer than twelve months. Working and calibration standards are prepared step by step dilution of stock solutions each day before analysis.

4.1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.2 Solutions

4.2.1 *Concentrated Nitric Acid* — Specific gravity 1.42 (*see* IS 264).

4.2.2 *Nitric Acid Solution* — 10 percent (*v/v*) in water.

4.2.3 *Concentrated Hydrochloric Acid* — Specific gravity 1.18 (*see* IS 265).

4.2.4 *Aqua-regia Solution* — Add concentrated nitric acid and concentrated hydrochloric acid in the ratio of 1:3 and carefully mix by stirring and store in polythene/ PTFE bottles. It is freshly prepared everyday for sample dissolution.

4.2.5 *Hydrofluoric Acid Solution* — 40 percent (*see* IS 10332).

CAUTION — All acids should be handled with care; extra precaution is required for handling hydrofluoric acid. Even when diluted this is extremely harmful to the eyes and skin; rubber gloves and safety visors should be used while using this acid. Any acid that touch the skin should be washed with copious amount of water and medical advice should be sought immediately. In case hydrofluoric acid is spilled on skin, saturated Boric acid solution should be applied after thorough washing with water.

4.2.6 *Boric Acid Solution* — 4.0 percent (*w/v*).

Dissolve 40 g of boric acid in 900 ml water, heat gently if required, cool and dilute to 1 000 ml.

4.2.7 *Sodium Hydroxide Solution* — 0.5N.

4.3 Stock Solutions

Prepare standard stock solutions from high-purity (99.9 percent or above) metal oxides or salts. Stock solutions of at least 100 ppm for each metal are convenient for preparation of dilute standards in the range from 0.01 to 10 ppm depending upon the sensitivity of the element and the instrumentation used.

4.3.1 *Standard Chromium solution*— [1 000 mg/l Cr(VI)].

Dissolve 3.735 0 g of potassium chromate (K_2CrO_4) in 250 ml of deionized water and dilute to 1 000 ml in a volumetric flask with deionized water and store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.2 *Standard Cobalt Solution*— [1 000 mg/l Co(II)].

Dissolve 4.937 9 g of cobalt nitrate [$Co(NO_3)_2$]in 200 ml of deionized water. Dilute to 1 000 ml in a volumetric flask with deionized water and store in a labelled polythene/PTFE bottles with leak proof screw cap.

4.3.3 *Standard Copper Solution*— [1 000 mg/l Cu(II)].

Dissolve 1.000 0 g of copper metal in 50 ml of 5M nitric acid. Dilute to 1 000 ml in a volumetric flask with deionized water and store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.4 *Standard Cadmium Solution*— [1 000 mg/l Cd(II)].

Dissolve 2.103 2 g of cadmium nitrate [$Cd(NO_3)_2$] in 250 ml of deionized water. Dilute to 1 000 ml in a volumetric flask with deionized waterand store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.5 *Standard Manganese Solution*— [1 000 mg/l Mn(II)].

Dissolve 3.607 7 g of manganese(II) chloride tetrahydrate ($MnCl_2.4H_2O$) in 50 ml concentrated hydrochloric acid (*see* **4.2.3**). Dilute to 1 000 ml in a volumetric flask with deionized water and store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.6 *Standard Nickel Solution*— [1 000 mg/l Ni (II)].

Dissolve 4.953 0 g of nickel nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$] in 100 ml of deionized water. Dilute to 1 000 ml in a volumetric flask with deionized waterand store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.7 *Standard Lead Solution* — [1 000 mg/l Pb(II)].

Dissolve 1.598 0 g of lead nitrate [$Pb(NO_3)_2$] in 100 ml of deionized water. Dilute to 1 000 ml in a volumetric flask with deionized water and store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.8 *Standard Zinc Solution*— [1 000 mg/l Zn(II)].

Dissolve 1.245 0 g of zinc oxide (ZnO) in 5 ml deionized water followed by 25 ml 5M hydrochloric acid. Dilute to 1 000 ml in a volumetric flask with deionized water and store in a labelled polythene/PTFE bottles with leak proof screw cap.

4.3.9 *Standard Lithium Solution*— [1 000 mg/l Li (I)].

Dissolve 6.109 0 g of lithium chloride (LiCl) in 100 ml deionized water. Dilute to 1 litre in a volumetric flask using deionized water and store in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.10 *Standard Vanadium Solution*

Dissolve 1.000 g of vanadium metal in 25 cm^3 of concentrated nitric acid (*see* **5.1.1**). Dilute to 1 000 ml in a volumetric flask with deionized water andstore in a labeled polythene/PTFE bottles with leak proof screw cap.

4.3.11 *Synthetic Coal Ash Solutions for Matrix Matching*

Synthetic coal ashsolutions are used to establish atomic absorption measurement conditions by matrix matching that takes care of interference caused by major coal ash constituents.

4.3.11.1 *Preparation of synthetic ash*

Synthetic ash is prepared from the constituents as mentioned in Table 1. The mixture is grounded to pass through a 212 micron IS sieve.

4.3.11.2 *Preparation of calibration solutions*

About 2.5 g of accurately weighed synthetic ash mixture is taken in PTFE pressure dissolution vessel. The screw cap is tightened immediately after adding 1.8 ± 0.2 ml of aqua regia solution and 3.0 ± 0.2 ml of hydrofluoric acid. The cups fixed in stainless steel cages are heated in an air oven at 160 ± 10°C for at least 2 h. The solution is allowed to cool and thereafter, 30 ml of 4 percent boric acid solution and 15 ml of water are added. Re-heating the pressure vessel in closed condition may be required if dissolution is not complete. The cooled solution is filtered through Whatman No. 42 filter paper. The solution is diluted to 1 litre using deionized water in a volumetric flask and labeled as Matrix Blank. Part of this solution is used as reagent blank. Calibration solutions of 10 ppm and 100 ppm are prepared form 1 000 ppm stock solutions (*see* **4.3**) step by step dilution

Table 1 Composition of Synthetic Ash
(*Clause* 4.3.11.1)

Major Constituents in Indian Coal Ash (1)	**Range** percent (2)	**Compounds for Synthetic Ash Standard** (3)	**Quantity** (g) (4)	**Concentration** percent, w/w (5)
SiO_2	45 to 67	Silicon dioxide	2.800	57.84
Al_2O_3	18 to 34	Aluminum trioxide	1.300	26.85
Fe_2O_3	3 to10	Ferric oxide	0.325	6.71
CaO	0.5 to 3	Calcium oxide	0.088	1.82
MgO	0.5 to 2	Magnesium oxide	0.075	1.55
TiO_2	1.8 to 2.5	Titanium dioxide	0.110	2.27
Na_2O	0.1 to 2	Sodium carbonate	0.093	1.92
K_2O	0.2 to 4	Potassium chloride	0.050	1.03
		Total	4.841	99.99

using deionized water. From measured quantities of the above solutions, calibration solutions for the analytes are prepared using the matrix blank solution as diluent to make upto 100 ml volume.

5 CERTIFIED REFERENCE MATERIAL OF COAL ASH

The use of appropriate certified reference materials (CRMs) is essential while checking the accuracy of the methods for the determination of trace elements in coal. CRMs of coal ash can be used where ashing of the coal is required as a part of the procedure. In absence of matrix standard, standard additions method may be employed to eliminate matrix interference.

6 APPARATUS

6.1 Atomic Absorption Spectrometer

The atomic absorption spectrometer shall have, in the absorption mode, a deuterium arc and other background correction facility.

6.2 Muffle Furnace

Gas or electrically heated in which a zone of constant and uniform temperature of 900 ± 10°C can be maintained. The heat capacity of the muffle furnace shall be such that, with an initial temperature of 900°C and with an empty crucible in position at least a temperature of 890°C is regained within 3 min when a charged crucible with stand is placed in its position.

6.3 Stirring Hot Plate and Bars

Operating temperature of 200 ± 5°C.

6.4 Bottles for Ash Dissolution and Storage of Atomic Absorption Spectrometer Measurement Solutions

Bottles made of polypropylene/polythene/PTFE with leak proof screw caps of 100 ml capacity.

6.5 Plastic Volumetric Flasks

Volumetric flasks made of polypropylene/polythene/PTFE of 100 ml and 1000 ml capacity with polypropylene screw closures.

6.6 Plastic Burettes

Plastic bottles of 1.1 litres to 1.2 litres capacity for storing composite, blank, calibration and reagent solutions

NOTE — In order to avoid contamination, the plastic materials are filled with nitric acid and allowed to stand for at least 48 h. The bottles are to be emptied and rinsed thoroughly with deionized water. Dry the bottles overnight in an oven set at 60°C.

6.7 Plastic Measuring Cylinder (*see* IS 10073)

6.8 Air Oven

Ventilated drying oven in which a constant and uniform temperature of 108 ± 2°C can be maintained.

6.9 Balance

An analytical balance capable of weighing upto 200 g with least count 0.1 mg.

6.10 Apparatus for Acid Dissolution of Samples

a) PTFE vessels with lid and steel casing.

CAUTION — At elevated temperatures, the closed PTFE vessels are under pressure (a few bars). The containers are not guaranteed under these conditions, and they may fail catastrophically, if safety precautions are ignored. Containers must be heated and cooled in a closed drying oven placed in a fume hood and these should not be peeked at unless these are at room temperature. Adequate safety precautions should be taken during their use. These containers tend to leak at 120°C unless inside a stainless steel cage.

b) Alternatively, a microwave digestion system, capable of being used in the temperature range of 240-260°C and maximum pressure in the range of 4-8 MPa may be used for sample dissolution.

7 SAMPLING AND SAMPLE PREPARATION

7.0 General

Samples are taken in accordance with

a) IS 436 (Part 1/Sec 1) for coal and lignite;

b) IS 436 (Part 2) for coke; and

c) IS 6491 for fly ash.

Special attention shall be taken in using sampling equipment to avoid contamination of the sample.

7.1 Preparation of Coal and Coke Samples

The samples obtained as above are received in sealed containers. Air-dried samples are grounded so that at least 98 percent shall pass through a 75 micron sieve. Special care shall also be taken to avoid contamination during milling.

7.2 Preparation of Fly Ash Samples

The fly ash sample is grounded in Agate or Mullite mortar to less than 50 micron top size. Special care shall be taken during the collection, preparation and storage of representative samples to avoid contamination.

7.3 Preparation of Sample Solution of Coal and Coke by Low Temperature Ashing

Coal or coke sample is converted into ash using low temperature ashing technique either by heating in an oxygen plasma asher (120-160°C) or in a muffle furnace using a silica dish at temperature of 450±25°C till constant mass.

NOTE — The low temperature ashing procedure for converting coal/coke into solution is not applicable for the estimation of volatile elements (namely Sb, Hg, Se, As, Ge, B etc). The low temperature ashing technique is preferred for better retention of the trace elements. Contact with metals such as stainless steel can result in contamination of the sample.

7.4 Acid Digestion of Low Temperature Ash and Fly Ash Sample

About 0.25 g of dry ash sample passing through 200 mesh sieve is accurately weighed and quantitatively transferred in a PTFE vessel (for pressure dissolution) of 100 ml capacity and moistened with 5 ml of deionized water. Then a mixture of 2 ml aqua regia and 2.5 ml hydrofluoric acid are added. The PTFE vessel is closed and tightly fixed in a stainless steel cage and then heated to about 160°C in an air oven for about 1 h. The vessel is then cooled to room temperature, 10 ml boric acid solution is added and re-heated in closed condition at 160°C for about 30 min. The resulting solution is cooled and filtered. Five ml of lithium chloride solution (2 percent) is added and the solution is diluted upto the mark in a 50 ml volumetric flask with deionized water.

NOTE — In place of lithium chloride, potassium chloride or cesium chloride may be used except for lithium.

8 PROCEDURE

8.1 Calibration

Solutions in the proportions described below are for typical samples as represented by Indian coals/coke/fly ash. Stronger or weaker concentrations may be required to establish suitable concentrations for elements of varying percentages outside the ranges of the typical sample. Plot the corresponding absorbance response for each calibration solution (as prepared in **4.3.11.2**) *versus* concentration and determine the linear range. Each analyte must have concentration ranges for standards compatible with the samples.

8.2 Duplicate Determination

Carry out parallel analysis in duplicate on separate test portions of the coal, coke or fly ash.

8.3 Blank Test and Sample Test

8.3.1 A blank test consisting of all reagents used in the method but omitting the test sample, shall be carried out at least in duplicate with each batch of test samples.

8.3.2 *Analysis of Test Solution*

Nitrous oxide-acetylene (N_2O-C_2H_2) flame is be used for Chromium and Vanadium and air-acetylene flame is used for Cadmium, Copper, Cobalt, Manganese, Nickel, Lead, Lithium and Zinc. Table 2 summarizes the parameters to be used for the individual elements.

NOTE — It is not possible to specify instrumental operating conditions because the instruments available vary significantly and incorporate alternative methods of atomization. Therefore, manufacturer's recommended operating conditions are to be followed.

Table 2 Instrument Parameters
(*Clause* 8.3.2)

Elements (1)	Wavelength nm (2)	Oxidant-Fuel and Flame Condition (3)
Cadmium	228.8	Air-Acetylene, Stoichiometric
Chromium	357.9	Nitrous oxide-Acetylene, Fuel Rich
Cobalt	240.7	Air-Acetylene, Fuel Lean Stoichiometric
Copper	324.8	Air-Acetylene, Stoichiometric
Lead	217.0	Air-Acetylene, Stoichiometric
Lithium	670.8	Air-Acetylene, Stoichiometric
Manganese	279.5	Air-Acetylene, Stoichiometric
Nickel	232.0	Air-Acetylene, Fuel Lean Stoichiometric
Vanadium	318.5	Nitrous oxide -Acetylene, Stoichiometric
Zinc	213.9	Air-Acetylene, Stoichiometric

8.3.3 Measure the absorbance of the test solution. From the plot of the absorbance response for each calibration solution *versus* concentration, determine the concentration of the element in test solution (C_{element}).

8.3.4 *Samples with Element Concentration Outside the Range*

Some samples may contain a higher level of an element than covered by the calibration solutions. In such a case, dilute a portion of the sample solution with the appropriate blank calibration solution so that the resulting solution is within the range of the calibration solutions. Dilute the test solutions to give an absorbance approximately equal to the mid range absorbance of the calibration plot. Record the dilution factor (F_{Dilution}). Measure the absorbance of the diluted test solution. From the plot of the absorbance response for each calibration solution versus concentration, determine the concentration of the test solution (C_{element}).

8.4 Minimization of Interference

The change in parameters (namely varying fuel/oxidant ratio, observation height and solution uptake rate) is made to minimize the interference with minimal change in sensitivity that is to achieve condition for getting maximum output absorbance signal.

9 EXPRESSION OF RESULTS

Moisture content and ash content are determined in accordance with the methods prescribed in IS 1350 (Part 1).

Calculate the moisture content of the sample as follows:

$$M_{\text{moist}} = \frac{(m_2 - m_3)}{(m_2 - m_1)} \times 100 \qquad \ldots(1)$$

where

m_1 = mass of the empty dish with lid, in g;

m_2 = mass of the empty dish with lid and sample, in g; and

m_3 = mass of the empty dish with lid and sample after heating, in g.

Calculate the ash content of the sample as follows:

$$M_{\text{Ash Sample}} = \frac{\left[(M_{\text{empty+sample}})_{\text{AA}} - m_{\text{empty}}\right]}{\left[m_{\text{empty+sample}}\right]} \times 100 \qquad \ldots(2)$$

where

$M_{\text{Ash Sample}}$ = mass of ash sample taken for the analysis, in g;

m_{empty} = mass of the empty dish, in g;

$m_{\text{empty + sample}}$ = mass of the empty dish plus sample, in g; and

$(m_{\text{empty+sample}})_{\text{AA}}$ = mass of the empty dish plus sample after ashing, in g.

From the calibration curve, the concentrations of the elements in the sample blank and test solutions are to be calculated. The element concentrations in the ash is given by the following equation:

$$C_{\text{element}} = \frac{100 \times (C_{\text{Test}} - C_{\text{Blank}}) \times F_{\text{Dilution}} \times M_{\text{Ash Sample}}}{(100 - M_{\text{moist}})} \qquad \ldots.(3)$$

where

C_{element} = element concentration in ash, in µg/g;

C_{Test} = element concentration in the test solution (or diluted solution), in µg/g;

C_{Blank} = element concentration in the blank solution, in µg/g;

$M_{\text{Ash Sample}}$ = mass of ash sample taken for the analysis, in g; and

F_{Dilution} = dilution factor if any.

10 PRECISION

10.1 Repeatability

Repeatability is based on the results obtained from duplicate determinations carried out over a short period of time but not simultaneously in the same laboratory by the same operator with the same apparatus. Repeatability is calculated from the results obtained on two different test solutions prepared from representative portions of the same ash sample. The results are calculated on dry coal/dry ash basis as specified in the section on Expression of Results to determine conformation to repeatability conditions mentioned in Table 3**.**

If the absolute value of the difference in results calculated to a dry coal basis is greater than repeatability values shown in Table 3, then the results are considered suspect.

10.2 Reproducibility

Reproducibility of results is based on the results carried out in different laboratories by different operators with different apparatus. Reproducibility is calculated from the results obtained on test solutions prepared from ashes from representative portions of the general analysis sample taken from the same bulk sample after the last stage of reduction. The results are calculated on dry coal/dry ash basis as specified in the section on Expression of Results to determine conformation to reproducibility conditions mentioned in Table 3.

If the absolute value of the difference in results calculated to a dry coal/ dry ash basis is greater than repeatability values shown in Table 3, then the results are considered suspect.

11 TEST REPORT

The test report shall contain the following information:

a) Complete identification of the sample tested.

b) Reference to the method used.

c) The trace element content expressed in µg of element per g.

d) The basis of reporting, for example, air-dry.

e) The analytical results and certified values of the CRM analyzed for quality assurance in the same batch as the sample.

Table 3 Precision Data for Trace Elements

(*Clauses*10.1 *and* 10.2)

Elements (1)	Concentration Range (µg/g), Dry Basis (2)	Repeatability (µg/g) (3)	Reproducibility (µg/g) (4)
Cadmium	0.01 to 20	1	2
Chromium	5-50	3	5
Cobalt	0.5 to 50	3	6
Copper	5 to 50	2	3
Lead	10 to 100	2	9
Lithium	20 to 100	2	5
	100 to 210	10	20
Manganese	< 50	3	6
	50 to 500	5	20
Nickel	< 5	1	3
	5 to 30	3	9
Vanadium	10 to 50	5	9
	50 to 100	6	20
Zinc	< 50	3	4
	50 to 100	8	17

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